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Oil-Infused Polymer Fiber Membranes as Porous Patches for Long-Term Skin Hydration and Moisturization

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Skin allergies and diseases, including atopic dermatitis (AD), affect millions worldwide. Current treatments for AD are often expensive, leading to a need for cost-effective solutions. Here, using fiber-based patches to maintain and increase skin hydration is explored, which helps treat eczema and AD. Nanofiber membranes are manufactured via electrospinning of eight different polymers: nylon 6 (PA6), polyimide (PI), poly(3-hydroxybuty-rate-*co*-3-hydroxyvalerate (PHBV), poly(L-lactide) (PLLA), polycaprolactone (PCL), and polystyrene (PS), and two molecular weights poly(vinyl butyral-*co*-vinyl alcohol-*co*-vinyl acetate) (PVB). Further, their morphology is examined through scanning electron microscopy (SEM), fibers, and pores diameter, wettability, and membrane thickness. Additionally, water vapor transmission rates (WVTR) are measured, and notably, skin hydration tests are conducted before and after using evening primrose oil-infused patches. The comparison and findings highlight the flexibility of electrospun patches, demonstrating their potential in maintaining skin hydration for 6 h and enhancing skin moisture, which are necessary in AD treatment. These insights, which focus on selecting the most effective performance patches, help improve skin moisture, leading to tailored treatments for AD, which can significantly impact the efforts to reduce healthcare costs and simplify skincare steps.

1. Introduction

Skin, the largest human organ, is the primary protective barrier against external threats.[1] However, it is often prone to diseases, environmental factors, aging, trauma, and genetics.[1,2] This vulnerability leads to an array of skin problems, such as eczema, referring to various skin inflammation conditions.[2] Atopic dermatitis (AD) is a specific type of eczema characterized by chronic inflammation and itching, often linked to a genetic predisposition to allergic conditions.[3] In 2017, AD affected ≈180 million people worldwide, and its impact on disability-adjusted life years is increasing.[4] Despite its widespread prevalence, treatments for AD are costly and often exceed those of chronic conditions like cardiovascular issues and diabetes.[5,6] Skin moisture is essential for maintaining skin health and integrity.[7,8] Nonpharmacological treatments, often incorporating natural extracts such as evening primrose oil, are widely used to create a protective barrier that decreases water loss and supports lipids in the skin.[9–12] The focus on natural oils aligns with the growing interest in holistic and natural health solutions and provides cost-effective options for everyday skin care and therapeutic applications.

Electrospinning has emerged as a revolutionary technique for creating porous membranes and scaffolds for tissue engineering,[13–15] wound healing,[16] tendon repair,[17] drug delivery, and treating skin issues.[18–21] Unlike existing treatments that often require frequent reapplication for consistent results, electrospun fibers can offer sustainable treatment.[19] The flexibility in producing fibrous membranes loaded with oils allows for greater control over release mechanisms and polymer selection.[19] These fibrous dressings are flexible, breathable, and easy to handle and maintain skin hydration by reducing water evaporation.[19,22] Moreover, the low production cost of electrospun membranes adds to their appeal.[23] The use of electrospun fibers in skin patches has brought attention to the importance of fiber size and arrangement.[24] Factors like the diameter and porosity of the fibers have proven to influence the effectiveness of a skin patch.[22,24] Furthermore, the size and arrangement of fibers within skin patches are critical in determining the spreading and release of oils or other therapeutic agents.[25] Evaluation of the usability and handling of elongated patches for bandage and dressing applications has further underscored the need for careful design and consideration of fiber properties.[24] These insights contribute to the ongoing development and optimization of skin patches as effective tools in medical and cosmetic applications. The use of different types of polymers adds a layer of complexity and opportunity in potential treatments. Key factors such as hy-

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Table 1. The average fiber diameter and the thickness of electrospun membranes used as patches for skin hydration tests.

Polymer	PA6	PVB(L)	PI	PVB(H)	PHBV	PLLA	PCL	PS
Thickness [μm]	30.0 ± 2.8	34.0 ± 2.7	28.1 ± 3.8	65.6 ± 5.2	30.0 ± 4.2	80.1 ± 9.8	89.0 ± 6.5	138.7 ± 4.1
Avg. fiber diameter [μm]	0.18 ± 0.02	0.39 ± 0.05	0.67 ± 0.07	0.87 ± 0.07	2.01 ± 0.29	2.68 ± 0.95	2.88 ± 1.26	5.94 ± 0.24

drophilicity play a significant role in determining the permeability, water sorption capacity, and degradability of materials, all of which influence the effectiveness of a skin patch.[26,27] This offers a level of control that can be instrumental in designing effective, tailored treatments for a variety of medical conditions, including skin disorders.

In this research, we investigate the potential of different electrospun fibers for designing a skin patch that can withstand real-world use. We used electrospinning to create eight different polymer membranes: nylon 6 (PA6), polyimide (PI), poly(3-hydroxybuty-rate-*co*-3-hydroxyvalerate (PHBV), poly(L-lactide) (PLLA), polycaprolactone (PCL), polystyrene (PS), poly(vinyl butyral-*co*-vinyl alcohol-*co*-vinyl acetate) (PVB) with high and low molecular weight, to develop skin hydration patches helping in AD treatment. By examining the fiber membranes' structure with scanning electron microscopy (SEM) and analyzing it with image processing, we explored how fiber and pore size can affect oil spreading and release, which is important in skin treatment. Additionally, we conducted water contact angle measurements and assessed membrane thickness to provide further insights into the material properties. Water vapor transmission rate (WVTR) was measured for the membranes, including those prewetted with evening primrose oil (EPO), to evaluate water exchange and breathability. Skin hydration tests were performed on ten volunteers using the electrospun membranes, and hydration was measured at 3 and 6 h after application. These comprehensive analyses contribute to understanding the potential of these new materials to enhance current methods applied in skin treatment. The findings of this study provide a significant step toward more affordable and efficient treatment, for example, eczema, aligning with the global efforts to enhance patient care and reduce healthcare costs.

2. Results and Discussion

2.1. Membrane Characterization

After manufacturing patches, we evaluated the morphology of the electrospun membranes and fibers. The SEM images presented in **Figure 1**, indicated a consistent and smooth fiber structure without any observable defects or beads for all samples. Fibers produced from PA6, PVB(L), PI, and PVB(H) exhibited average fiber diameters of less than 1 μm. In contrast, the other four polymers—PHBV, PLLA, PCL, and PS—exhibited fiber diameters ranging from 1 to almost 7 μm; see Figure 1i and **Table 1**. Additionally, the pore analysis in the electrospun membranes, which includes the pore area and the average size of the pores, is shown in Figure 1j. Pore area is a crucial attribute as it influences the overall porosity of the mats, thereby affecting their permeability and breathability. The highest pore area percentages were exhibited by PVB(H) (49.8%) and PI (49.7%) membranes, suggesting a potentially higher permeability compared to the other samples. The PS membrane, on the other hand, had the lowest pore area at 33.8%. There was a significant variation in the size of the pores across the materials. PS had the largest average pore size at 105.3 ± 12.8 μm, followed by PLLA (51.2 ± 2.1 μm) and PCL (34.1 ± 3.2 μm). PA6, conversely, had the smallest average pore size of 0.22 ± 0.04 μm, which was considerably smaller than the other materials. The pore area and pore size characteristics showcase the porous nature of the electrospun fiber mats. The difference in these properties across the diverse materials is likely due to the variations in fiber diameter and the electrospinning process parameters.[28,29] Porosity, pore shape, and pore size significantly impact the functionality and potential applications of these membranes.[30,31]

Following a thorough examination of the membrane's morphology, we assessed the purity and molecular structure by conducting Fourier-transform infrared spectroscopy (FTIR) measurements. These measurements were in accordance with the specifications provided by the manufacturers; see Figure S1 (Supporting Information). Next, the wetting properties of all electrospun membranes were examined through water contact angle measurements, as depicted in Figure 1k. The water advancing contact angles for PVB(L), PI, PVB(H), PHBV, PLLA, PCL, and PS predominantly ranged from 110° to 140°, demonstrating a primarily hydrophobic character. This behavior could make these materials favorable for applications where moisture resistance is a key requirement.[32] On the other hand, PA6 exhibited a significantly lower contact angle of 60.4°, indicating hydrophilic behavior. This trait suggests a high affinity for water and could render PA6 more suitable for applications requiring water absorption or permeability.[33] These findings generally align with previously reported results, as similar water contact angles were reported for PA6, PS, and PCL.[34] For electrospun PVB, hydrophobic properties were reported, with a contact angle range of 130°–135°, consistent with earlier observations.[35] Comparably, a hydrophobic result of 143° was reported for PI fibers, aligning with our findings.[36] The hydrophobic behavior of electrospun PLLA was also indicated with a similar contact angle to what we observed.[37] In summary, the water contact angle measurements confirm the predominantly hydrophobic nature of most of the studied electrospun membranes, with PA6 demonstrating distinct hydrophilic behavior.

Further, the WVTR for electrospun membranes, both untreated and oil-treated ones, are shown in Figure 1l. All untreated meshes, thus as produced electrospun membranes, demonstrated similar WVTR, highlighting no significant variances in their initial states. However, a marked decrease in WVTR was evident in all membranes following oil treatment. A significant reduction in WVTR of 70.5% was noted for the hydrophilic PA6 upon oil treatment. Similarly, oil-treated hydrophobic samples exhibited substantial reductions in WVTR. Specifically, WVTR for

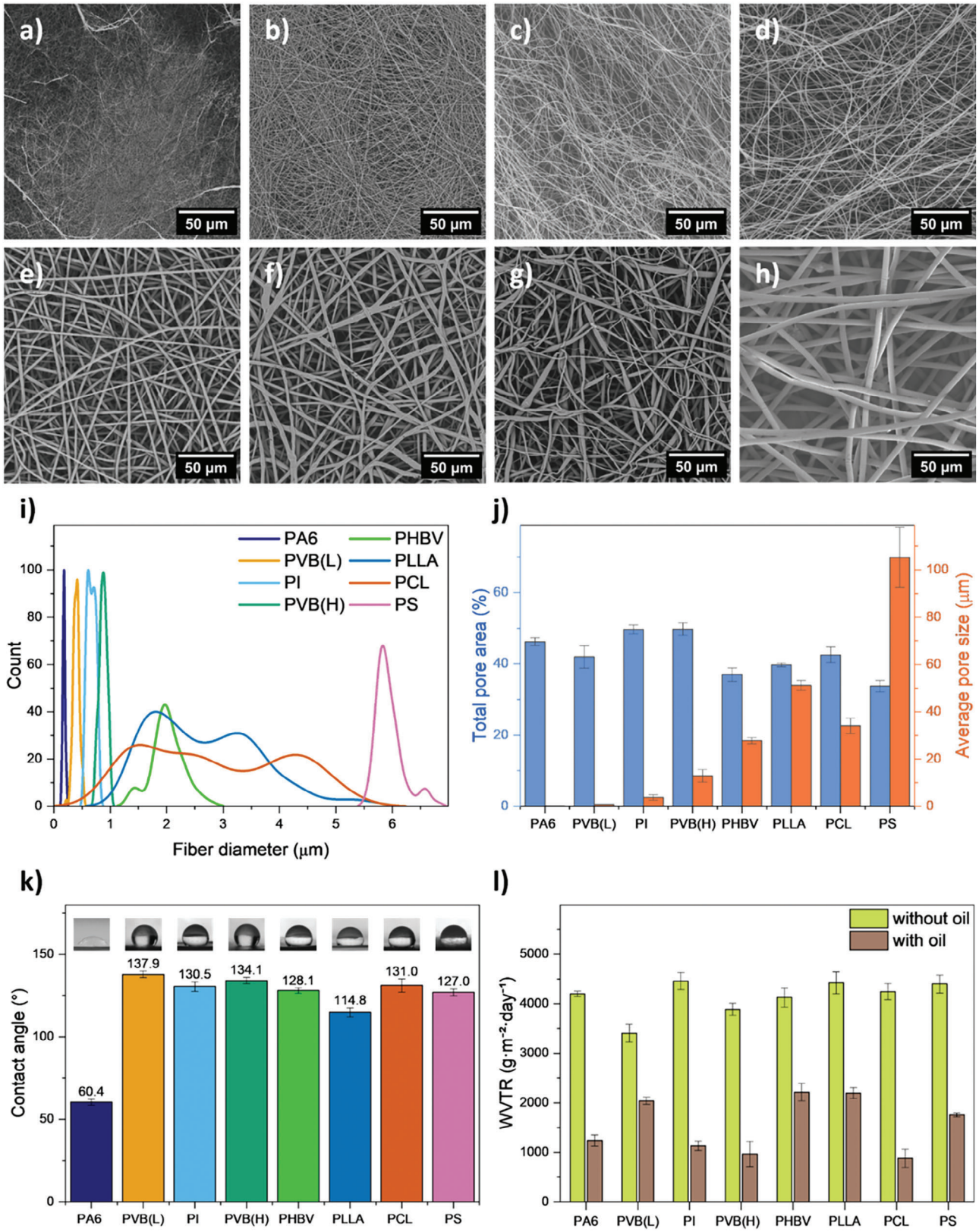

Figure 1. SEM images of membranes from a) PA6, b) PVB(L), c) PI, d) PVB(H), e) PHBV, f) PLLA, g) PCL, and h) PS. Together with i) distribution of fiber diameter and j) total area and pore size for a given polymer, k) wetting contact angle results with an exemplary image of a water droplet and indicated the average values, and l) the column chart showing water vapor transmission rate with and without oil through the electrospun patches.

PI, PVB(H), and PCL decreased by 74.6%, 75.1%, and 79.3%, respectively. A more moderate decrease between 40% and 60% was recorded for PVB(L), PLLA, PCL, and PS. These observations suggest that oil treatment significantly mitigates the WVTR of electrospun membranes, irrespective of their wetting properties. The application of oil seems to create a barrier that obstructs the transmission of water vapor, and high porosity enables a more thorough oil barrier. The data indicate that hydrophilicity does not affect the gas exchange between the beaker and the environment for materials with a porosity of >30%. The data exhibited consistency with previously published results for the PA6 membrane.[38] However, for PS, our findings diverged from previous literature, noting a substantial decrease in WVTR after oil application—an observation absent in prior studies.[38] PI showed similar values to previously reported data, although our results presented a more notable decrease in WVTR.[25] For PVB(L) and PVB(H), our values were in line with those reported previously, with slight variation after oil application.[24] In PCL patches, the described WVTR for a dry PCL/chitosan scaffold is similar.[39] Importantly, adding oil to electrospun patches significantly decreases their WVTR. Here, the porosity of these membranes plays a critical role in gas exchange.[40,41]

2.2. Skin Hydration

The characteristics of adherence, flexibility, and conformability exhibited by the skin hydration patches when applied to volunteers play a crucial role in determining their effectiveness and practical utility. **Figure 2**a provides images of the patches applied to volunteers at the beginning of the skin hydration experiments. These images highlight the initial conditions and demonstrate the variations in the behavior of the different fiber meshes in a real-world application. The initial skin moisture levels were measured prior to conducting the skin hydration experiments with the electrospun fiber mats; see Figure 2b. The combined results for the 3 and 6 h tests show a range of hydration levels across the subjects. The initial condition for the test exhibited hydration levels ranging from 11.7% to 33%, with standard deviations indicating relatively low variation for each individual. It is important to note that an initial skin hydration level below 30% is commonly associated with a dry skin condition.[42] In our study, all subjects exhibited initial hydration levels below or just around this threshold. This characteristic is significant as dry skin is one of the common symptoms associated with skin problems such as eczema and especially AD. By comparing the subsequent hydration levels with the initial conditions, we can assess the effectiveness of the fiber mats in maintaining or improving skin moisture levels. The hydration levels of the electrospun patches were evaluated through the measurement of Δ hydration, representing the change in hydration compared to the initial measurement on the skin of a volunteer before the treatment. All the results obtained at 3 and 6 h under dry conditions and when treated with oil on the membrane are presented in Figure 2c.

Under dry conditions, most fiber mats exhibited a negligible-to-moderate change in hydration levels. Notably, PS, PVB(H), and PA6 presented marginal increases in hydration. PVB(L) exhibited only a small increase in hydration at the 3 h mark, followed by a decrease at 6 h. Meanwhile, PI, PHBV, PLLA, PCL, and the control showed moderate increases in skin hydration. These results suggest that the skin moisture can be controlled via the usage of a specific polymer membrane. In contrast to the reference samples, under oil-treated conditions, all the materials exhibited substantial hydration enhancement at both time points. The magnitude of this increase—expressed as Δ hydration values—was greater than that observed under dry conditions. PVB(L) and PHBV demonstrated the highest increase in hydration at the 3 h mark, while PI exhibited the highest value at 6 h. Notably, for PA6 and PCL, we observed high values that remained the same for both measured periods, ≈16.5% and ≈18.5%, respectively. These findings provide substantial evidence that oil treatment enhances the hydration capabilities of the fiber membrane significantly compared to its dry counterparts. And that oil delivery through the patches allows a controlled release, securing the prolonged effects in skin hydration level.[43]

Interestingly some material-specific behaviors were observed during the study. PI and PCL yielded the most satisfactory results, indicating their potential for prolonged moisturization up to 6 h and real-world applications. Both materials were easy to handle, adhered well to the skin throughout the whole duration of the study and yielded high results in skin hydration tests; see Figure 2a. Patches based on PVB(L), PVB(H), and PA6 were also easily manageable, maintained their shape once placed on the skin, and demonstrated good skin conformity and adhesion throughout the 6 h experimental period. Without oil treatment, PA6 and PVB(L) exhibited a reduction in hydration, indicating that oil passing through the pores in the patches has a moisturizing effect. All other meshes without oil treatment induced a slight moisture increase, likely due to an obstruction in water release from the skin, as also suggested by WVTR data shown in Figure 1l. As for tests with oil, PVB(H) performance improved over time by an extra 5.6% increase from 3 to 6 h. The skin tests demonstrated that PHBV and PLLA patches were rigid and had poor conformity to the body. It led to noncontinuous oil release and improper skin coverage due to the gaps between the patch and the skin. These issues, along with the detachment of the mats during the experiment, caused the hydration levels to drop sharply when tested after 6 h. PS mats did not cover the skin adequately due to their highly 3D structure and tendency to tangle and clump.[44,45] Despite adhesive tape application to secure the mat, hydration levels remained comparable to the control, indicating poor attachment to the skin.[27] Similarly, an improvement in skin moisture levels for PA6 and PS was observed. However, using a higher volunteer count allowed us to reveal that PS mesh is not stable enough for a prolonged study of up to 6 h and is prone to tangling and clumping.[38] For PS, the thickness of the electrospun membrane is also higher; see Table 1. For PVB patches, the skin hydration increase is confirmed.[24] Notably, we observed a similar increase in skin moisture; however, the results after 6 h with oil show a reversed trend where the PVB(H) was better at keeping the skin moisture. In the case of patches made of PI, we obtained the results in line with a study performed on a smaller group of volunteers showing hydration improvement during a 6 h experiment.[25] A good agreement with the literature can also be found for PHBV, where the hydration increased by about 20% for each of the five tested volunteers.[46] Moreover, PCL membranes were also reported as skin patches loaded with hemp oil, where

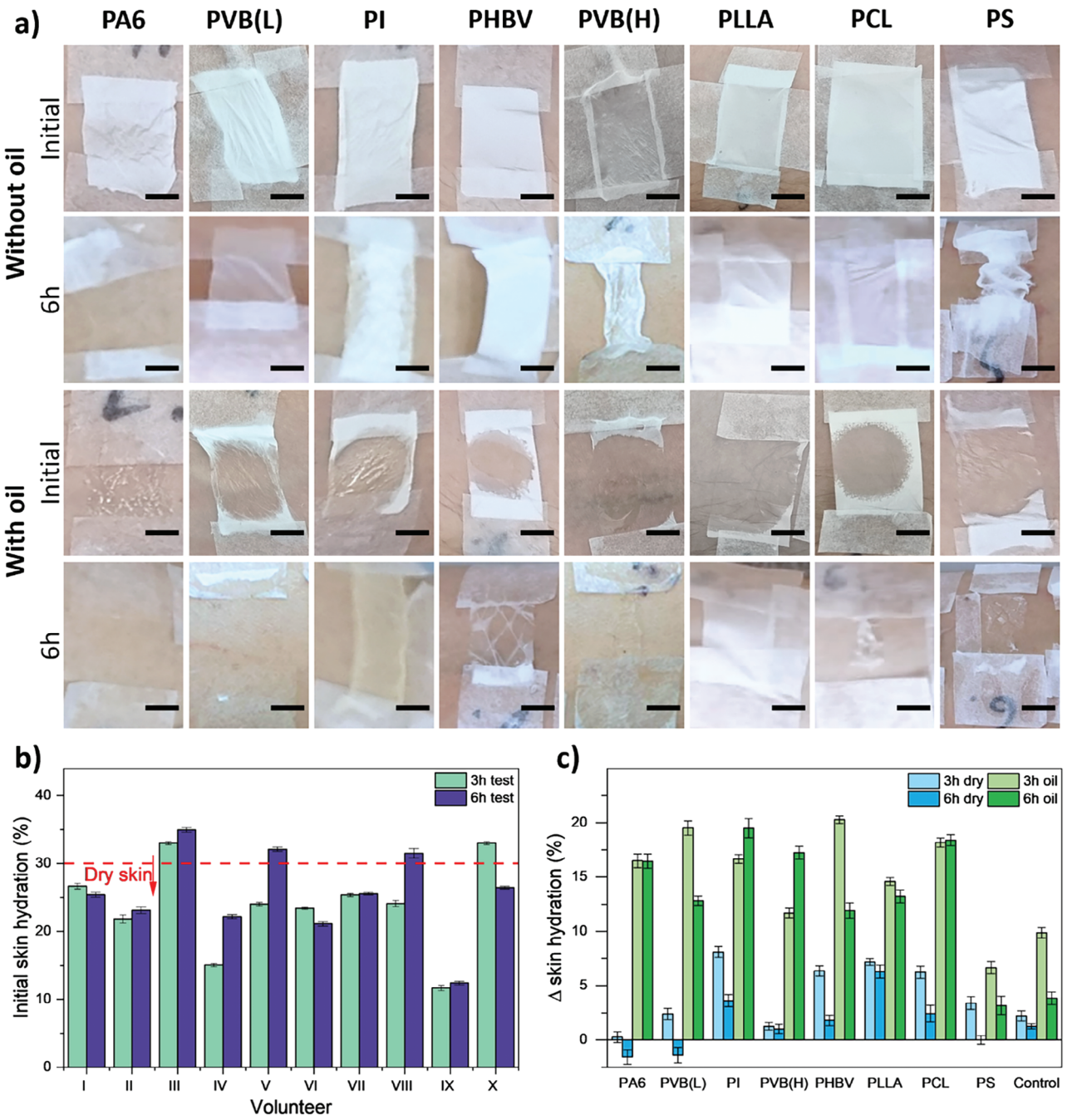

Figure 2. Skin hydration test results on eight types of patches: a) patches on volunteers at the beginning and after 6 h of skin hydration experiments; patches are based on polymers indicated on the top of images. b) Initial skin hydration of volunteers; the dashed line on the graphs indicates the skin hydration level for dry skin, which is below 30%. c) Δ skin hydration after 3 and 6 h. The control test is performed on the skin without any patch. *Scale bar denotes 0.5 cm.

around 20% skin hydration increase was reported for three volunteers.[22] Interestingly, different oil type led to a similar skin hydration increase, as we show in Figure 2c. For PLLA patches, we observed a third lowest result after 3 h (14.6%), with PVB(H) and PS being worse (11.7% and 6.7%); however, after 6 h, the results for PLLA (13.2%) exceeded marginally those from PVB(L), PHBV, and PS (12.8%, 11.9%, and 3.2%, respectively).

Differences in skin hydration between 3 and 6 h are tied to oil transport in porous media where wetting properties, pore area, size, and shape, together with fiber diameter, play an important role.[19] A study on PVB has shown that small fibers allow faster oil transport through the porous membrane.[24] For overnight treatment, a slower release of oils is more beneficial, as indicated by 6 h test results. Regardless, in our study, the amount of oil was

adequate to saturate all the membranes and provided a barrier for up to 6 h in all cases. This comprehensive study demonstrated various electrospun membranes' application as skin patches able to treat dry skin that is observed in AD cases and keep a high skin hydration level. The presented findings illustrated that elevated skin hydration levels depended on the polymers used to design for the patches and treatment conditions. The findings underscore the importance of material composition and geometry in determining the performance of electrospun membranes as skin hydration aids. Furthermore, applying oil on the mats significantly enhanced their hydration promotion capabilities.

3. Conclusion

Our comprehensive study on various electrospun fiber membranes revealed valuable insights into their potential applications. Based on our analysis of the morphology of the fibers, we found that the fiber diameter varied across different materials, with PA6, PVB(L), PI, and PVB(H) having diameters of less than 1 μm, while PHBV, PLLA, PCL, and PS fibers exhibited larger diameters reaching almost 6 μm. The pore characteristics suggested variations in potential permeability across the different materials. Contact angle measurements revealed the primarily hydrophobic nature of most materials, with the exception of PA6, which exhibited hydrophilic behavior. The WVTR measurements suggest that oil treatment significantly reduces the WVTR for all membranes, irrespective of their wetting properties. The results indicate the potential role of porosity in gas exchange, especially at higher porosities. In the skin hydration tests, the oil-treated fiber mats showed significant enhancement in hydration compared to the dry conditions, irrespective of the type of fiber. Moreover, the behavior of these mats was material specific, and their application performance varied, suggesting the importance of material choice for specific applications. The hydration capabilities of fiber-based membranes were significantly enhanced with EPO. The best result after 3 h, increased hydration by 20.3%, was for PHBV membrane, and after 6 h, was recorded for PI patch (19.5%). Such a high result after 6 h makes the material a good candidate for overnight treatment. Notably, the best overall hydration for both 3 and 6 h was recorded for PCL of 18.2% and 18.4%, respectively. Such a patch would be well suited for a patch designed for general use. Generally, the hydrophobic membranes having a thickness of around 30 μm with a fiber diameter below 1 μm are ideal candidates. In the case of thicker patches, with a thickness of 80 μm, the larger pores reaching an order of magnitude higher average pore size maintained their shape once placed on the skin and kept the skin moisturized for 6 h. The study findings offer valuable insights for the design and selection of materials for applications where moisture management is critical, such as in the skin hydration and health sectors.

4. Experimental Section

Electrospinning: Polymer membranes from all polymers tested were produced using electrospinning. The electrospinning process was carried out with climate control using the apparatus IME Technologies (Waalre, The Netherlands) for all polymers except PLLA, which was produced using the SKE E-FIBER EF 100 equipment (SKE Research Equipment, Italy). Depending on the specific material, the electrospun fibers were created using voltages ranging from 11 to 20 kV and a nozzle-to-collector distance of 15–20 cm. Detailed information on solution preparation and electrospinning parameters for each sample is given in Table S1 (Supporting Information).

Membrane Morphology: The morphology of the electrospun membranes was examined with SEM. The electrospun membranes were carefully cut into appropriate sizes and mounted on microscopy stubs using conductive double-sided adhesive tape. The surfaces were then sputter-coated (Quorum Q150RS, Quorum Technologies Ltd., UK) with a 10 nm layer of Au. The SEM analysis was conducted using Phenom ProX (Thermo Fisher Scientific, USA) with an accelerating voltage of 10 kV and a working distance of 3–7 mm. The acquired SEM images were analyzed using ImageJ software (v. 1.53d, USA) to evaluate morphological parameters such as fiber diameter and membrane porosity. The average fiber diameter was calculated from 100 measurements taken across multiple images. After the images were binarized using the threshold function, porosity was measured using ImageJ software's analyze particle function.

The Thickness of the Membranes: An optical microscope (Axio Imager, Zeiss, Germany) was employed to measure the thickness of the polymer mats. The thickness was determined by measuring the vertical distance between the bottom substrate and the top surface of the fiber mat. Five measurements were taken from various sample regions, and their average value was calculated.

Chemical Composition of Membranes: Molecular structure and purity of all membranes were tested using FTIR (Nicolet iS5, Thermo Fisher Scientific, USA) with OMNIC 9 software (version 9.12.928, Thermo Fisher Scientific, USA). Membranes were analyzed in attenuated total reflectance (ATR) mode. For each sample, 64 scans with a resolution of 4 cm^{-1} in the 600–4000 cm^{-1} range were performed. These measurements were conducted on clean electrospun fibers before the infusion of oil.

Wettability: The mats' water contact angle (θ) was determined using the direct method. Deionized (DI) water (Spring 5UV purification system Hydrolab, Straszyn, Poland) was used as the measuring liquid. A volume of 3 μL of DI water was applied to each electrospun sample using an automatic pipette (HTL 0.5–5 μL, Acon, USA). The test was conducted at a temperature of 22 °C and relative humidity (RH) = 32%. Images of the droplets were captured within 4 s of deposition using a Canon 700d camera with a Canon EF-S 60 mm *f*/2.8 Macro USM lens (Canon, Japan). For each material, the contact angle was measured for ten drops using ImageJ software, and the average values and standard error were calculated.

Water Vapor Transmission Rate: The WVTR test was adapted from a previously reported procedure and applied to PA6, PVB(L), PI, PVB(H), PHBV, PLLA, PCL, and PS membranes.[24] The experimental procedure involved filling the beakers with 5 mL of distilled water, followed by tightly placing the electrospun membranes on top. Subsequently, the samples were weighed and submerged in a water-filled bath maintained at 37 °C and a humidity range of 30–32% for 24 h. After the experiment, the samples were reweighed using a precision scale (Ohaus, Switzerland) to determine the water loss in relation to the initial weight. Two types of samples were prepared to investigate the influence of oil application on WVTRs: dry membranes placed on the beakers and membranes prewetted with 25 μL of evening primrose oil. The average WVTRs and standard error were calculated based on three measurements for each sample type.

Skin Hydration Test: EPO was used to assess the effectiveness of electrospun mats made from PA6, PVB (L), PI, PVB (H), PHBV, PLLA, PCL, and PS membranes as skin hydration patches. Initial skin hydration measurements were conducted using a Corneometer (Hydro Pen HP 10, Courage+Khazaka electronic GmbH, Germany) on a group of ten volunteers: males and females aged 24–42 years, representing a range of dry to moisturized skin. Rectangular patches (2 × 3 cm) were prepared and attached to the skin using paper tape (Dr. Max, Eurosirel S.p.A., Italy). Dry and oil-treated patches were prepared for each type of polymer membrane, with 25 μL of EPO applied between polymer fibers. The skin hydration was measured at 3 and 6 h after patch application. Five measurements were taken for each sample at each time point to calculate the average skin hydration before and after the experiment, along with the standard error. The hydration test was carried out following an already existing procedure, and all ten volunteers who participated in the study carried out regular office work activities during the testing period.[24] The patches on human

skin were conducted on volunteers in compliance with ethical guidelines for human testing of cosmetic products set forth by the EU Council Directive (76/768/EEC) and the World Medical Association Declaration Helsinki (1964-1975-1983-1989-1996).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

atopic skin, eczema, electrospinning, hydration, patches, polymer fibers

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